

Changes in Surface Chemistry
of Silicon Carbide (0001)
Surface With Temperature
and Their Effect on Friction

Kazuhisa Miyoshi and Donald H. Buckley

NOVEMBER 1980

LIBRARY COPY
DEC 8 2 1980
LANGLEY RESEARCH CENTER
LIBRARY, NASA
HAMPTON, VIRGINIA

NASA

3 1176 00509 0130

NASA Technical Paper 1756

Changes in Surface Chemistry
of Silicon Carbide (0001)
Surface With Temperature
and Their Effect on Friction

Kazuhisa Miyoshi and Donald H. Buckley
Lewis Research Center
Cleveland, Ohio



National Aeronautics
and Space Administration

**Scientific and Technical
Information Branch**

1980

Summary

An investigation was conducted to examine the effects of temperature on the surface of silicon carbide and the friction behavior of a silicon carbide (0001) flat surface in sliding contact with a polycrystalline iron pin specimen. Surface pretreatments examined included (1) bombarding the surface with argon ions for 30 minutes at a pressure of 1.3 pascals, (2) heating it at 800° C for 3 hours in a vacuum of 10^{-8} pascal, and (3) heating it at 1500° C for 3 hours in a vacuum of 10^{-8} pascal. Friction experiments were conducted in a vacuum of 10^{-8} pascal, and Auger emission spectroscopy was used to assess the presence of silicon and carbon, and the form of the carbon, on the silicon carbide surface after surface bombardment and heating.

The surfaces of silicon carbide bombarded with argon ions or preheated to 800° C revealed the main silicon peak and a carbide type of carbon peak in the Auger spectra. The surfaces preheated to 1500° C revealed only a graphite type of carbon peak in the Auger spectra, and the silicon peak diminished to a barely perceptible amount. The surfaces of silicon carbide preheated to 800° C gave a 1.5 to 3 times higher coefficient of friction than did the surface of silicon carbide preheated to 1500° C. The coefficient of friction was markedly dependent on orientation, and it was lower in the $\langle 1120 \rangle$ direction than in the $\langle 1010 \rangle$ direction. That is, the coefficient of friction was lower in the preferred crystallographic direction of slip.

Introduction

The high strength and excellent oxidation and creep resistance of silicon carbide make it an extremely important material for high-temperature mechanical and electronic applications in severe environments. It is used, for example, in stable high-temperature semiconductors, in gas turbine blades, in turbine ceramic seals, and as an abrasive for grinding (refs. 1 and 2).

In grinding, as a result of the extremely small chip size and the high wheel speed involved, instantaneous temperatures and pressures at the tip of a silicon carbide abrasive particle are extremely high (ref. 3). In addition, the freshly formed surfaces of silicon carbide are highly reactive with metals. The

fundamental tribological properties of silicon carbide in contact with metals are not, however, clearly understood.

The authors of this report have conducted experimental work at room temperature to gain an understanding of the surface of silicon carbide and its adhesion and friction properties (refs. 4 and 5). These properties depend strongly on the surface characteristics of silicon carbide. In turn the surface characteristics of silicon carbide are strongly affected by temperature (refs. 6 and 7). For example, an increase in temperature in vacuum can cause graphitization of the carbon and depletion (by evaporation) of the silicon. The tribological behavior of silicon carbide at high temperatures in vacuum is therefore important. The knowledge gained from such studies can assist us in achieving a much better understanding of the surfaces and characteristics of silicon carbide.

The objective of the present investigation was to examine the effects of temperature on the surface chemistry of silicon carbide and the friction behavior of silicon carbide sliding against iron. The orientation of silicon carbide was also examined relative to its effect on friction. The silicon carbide surface was pretreated (1) by bombarding it with argon ions for 30 minutes at a pressure of 1.3 pascals, (2) by heating it at 800° C for 3 hours in a vacuum of 10^{-8} pascal, or (3) by heating it at 1500° C for 3 hours in a vacuum of 10^{-8} pascal. The *in situ* friction experiments were conducted with the single-crystal silicon carbide (0001) flat surface in contact with a pin of polycrystalline iron at room temperature and at temperatures to 1200° C. Experiments were conducted with loads to 0.30 newton at a sliding velocity of 3×10^{-3} meter per minute in a vacuum of 10^{-8} pascal (10^{-10} torr). *In situ* Auger emission spectroscopy analysis was used to monitor the surface chemistry.

Materials

The single-crystal α -silicon carbide used in the experiments was a 99.9-percent-pure compound of silicon and carbon. The crystal was grown by a carbon arc method. Silicon carbide has a hexagonal, close-packed crystal structure, with the most commonly occurring unit cell dimensions being $a = 0.30817$ nanometer and $c = 1.51183$ nanometers

(table I, fig. 1, and ref. 8). The crystal structure of silicon carbide can be considered in terms of a hexagonal unit cell containing two interpenetrating, close-packed atomic arrays—one of silicon and the other of carbon displaced by one-quarter of a layer spacing along the c axis. The silicon atoms thus occupy the tetrahedral locations in the array of carbon atoms, and vice versa (refs. 9 and 10). Hence the basic unit of the structure can be considered to be a plane of a tetrahedron, arbitrarily SiC_4 or CSi_4 .

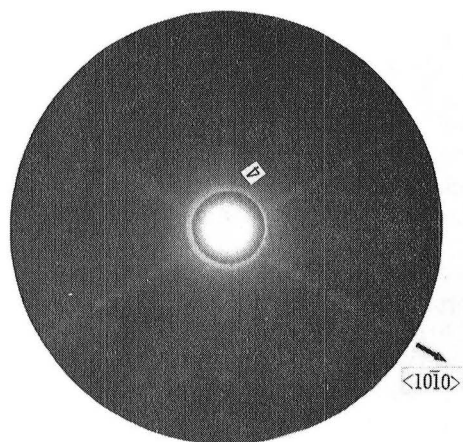
The (0001) plane was nearly parallel to the sliding surfaces examined herein. X-ray back-reflection Laue photographs were taken to establish the exact bulk orientation of the crystals after the crystals had been polished with a diamond powder (3- μm diam) and then an aluminum oxide powder (1- μm diam). A Laue photograph is shown in figure 1. Specimens were within $\pm 2^\circ$ of the low index (0001) plane. The silicon carbide samples were in the form of flat platelets and had a mean surface area of about 70

TABLE I.—COMPOSITION AND CRYSTAL
STRUCTURE OF SINGLE-CRYSTAL
SILICON CARBIDE

Composition ^a :	
Silicon, percent.....	66.6
Carbon, percent.....	33.3
Oxygen, ppm	< 500
Boron, ppm	< 100
Phosphorous, ppm.....	< 200
Others, ppm.....	< 0.1
Structure ^b :	
Interatomic distance, nm:	
a	0.30817
c	1.51183
Lattice ratio, c/a	4.9058

^a Manufacturer's analysis.

^b Ref. 8.



Backreflection Laue X-ray photograph of
{0001} plane

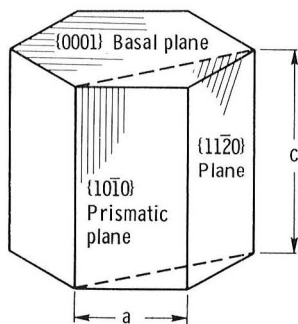
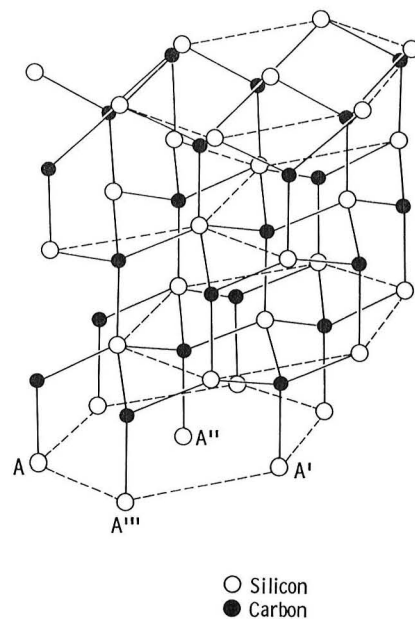
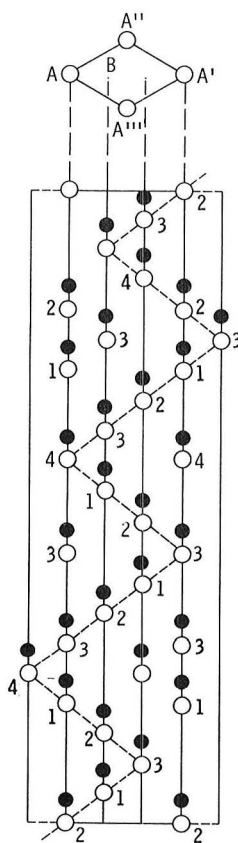


Diagram of hexagonal crystal



○ Silicon
● Carbon

Structure of α -SiC

Figure 1. - Crystal structure of silicon carbide.

square millimeters or more. The roughness of the silicon carbide surfaces measured by surface profilometer was 0.1 micrometer for the maximum height of irregularities.

The iron was electron-beam-zone refined and was polycrystalline and 99.99 percent pure. The radius of the iron pin specimen was 0.79 millimeter.

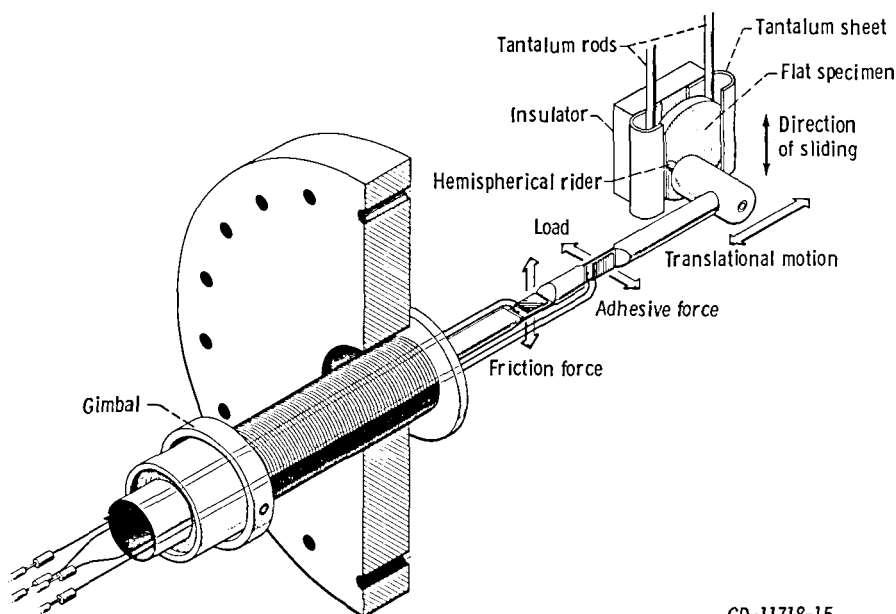
Apparatus

The apparatus used in the investigation was mounted in an ultra-high-vacuum system. The apparatus is capable of measuring adhesion, load, and friction, as is shown schematically in figure 2. The vacuum system also contains an Auger emission spectrometer (AES) for surface analysis. A gimbal-mounted beam is projected into the vacuum system. The beam contains two flats machined normal to each other with strain gages mounted thereon. The iron metal pin is mounted on the end of the beam. The load is applied by moving the beam normal to the flat and is sensed with the strain gage.

The vertical sliding motion of the pin along the flat surface is accomplished through a motorized gimbal assembly. Under an applied load the friction force is measured during vertical translation by the strain gage mounted normal to that used to measure load. This feature was used to determine the coefficient of friction at various loads.

Experimental Procedure

To heat the silicon carbide specimens by resistance heating, tantalum thin-film coatings were applied to the back surfaces of five silicon carbide crystals in a commercial radiofrequency diode apparatus. The sliding surfaces of the silicon carbide specimens to be used in the friction experiments were then polished and cleaned with diamond powder (3- μ m diam) and with aluminum oxide powder (1- μ m diam). The back of each specimen was attached to tantalum rods with tantalum supporting sheets (fig. 2). The tantalum-coated surface of the specimen was directly in contact with the rods. The flat and pin specimens were rinsed with 200-proof ethyl alcohol, just before being placed in the vacuum chamber. The specimens were placed in the vacuum chamber as shown in figure 2, and the system was evacuated and subsequently baked out to obtain a pressure of 10^{-8} pascal. When this pressure was obtained, argon gas was bled back into the vacuum chamber to a pressure of 1.3 pascals, and the pin specimen was argon ion bombarded for 30 minutes at -1000-volt direct current potential. After the bombardment of the pin specimen was completed, the vacuum chamber was reevacuated. Then surface pretreatments were conducted *in situ* on the silicon carbide flat specimens. The surface pretreatments examined included (1) ion bombarding the silicon carbide surface with argon ions at



CD-11718-15

Figure 2. - High-vacuum friction and wear apparatus.

– 1000-volt direct current potential in the vacuum chamber at a pressure of 1.3 pascals for 30 minutes, (2) heating the surface at 800° C at a working pressure of 10⁻⁸ pascal for 3 hours, and (3) heating it at 1500° C at 10⁻⁸ pascal for 3 hours.

In the first surface pretreatment the crystal was in the as-received state after it had been baked out in the vacuum system at 10⁻⁸ pascal. Argon gas was then bled back into the vacuum chamber to a pressure of 1.3 pascals, and the flat silicon carbide specimen was argon ion bombarded for 30 minutes with a direct current potential of – 1000 volts. AES spectra of the silicon carbide flat surface were obtained to examine the surface chemistry before and after ion bombardment.

The second surface treatment involved the crystal in the as-received state after bakeout in the vacuum chamber. The silicon carbide surface was, however, in this case resistance heated to 250° C and was held at this temperature for as long as 3 hours at 10⁻⁸ pascal. After heating, the specimen was cooled to room temperature. Auger spectra of the specimen were obtained before and after heating.

Subsequently specimens were heated to 400°, 550°, 700°, and 800° C; and Auger analyses after cooling were conducted in the same manner as that to 250° C with heating times to 3 hours at each temperature in a vacuum of 10⁻⁸ pascal. After being heated to 800° C and cooled to room temperature the silicon carbide specimen was next reheated to 800° C for friction experiments.

The third surface treatment also employed the crystal in the as-received state after bakeout in the vacuum chamber. In this instance the surface was heated from room temperature to 1500° C in 150 degree C intervals with successive heatings of 1 to 3 hours at each temperature. Auger spectra were obtained after heating at each temperature, as already mentioned. After being heated to 1500° C and cooled to room temperature the silicon carbide specimen was next reheated to 1200° C for friction experiments. The power for resistance heating of the silicon carbide specimen was supplied through the tantalum rods and film coating by a precisely regulated direct-current output that was adjustable over a wide range.

The temperature of the silicon carbide surface was measured with a conventional thermocouple in direct contact with the surface of the silicon carbide specimen in a vacuum. Friction experiments *in situ* were then conducted with the surface-treated silicon carbide specimens over a temperature range from room temperature to 1200° C.

Loads of 0.05 to 0.3 newton were applied to the pin-flat contact by deflecting the beam of figure 2. Both the load and the friction force were

continuously monitored during a friction experiment. Sliding velocity was 3×10^{-3} meter per minute with a total sliding distance of about 2 or 3 millimeters. All friction experiments in vacuum were conducted with the system evacuated to a pressure of 10⁻⁸ pascal. The coefficients of friction reported herein were obtained by averaging three to five measurements. The standard deviations of most data were within 3 to 4 percent of the average value.

Results and Discussion

Auger Analysis of Silicon Carbide Surfaces

Argon sputter-cleaned surface.—An Auger emission spectroscopy spectrum of the single-crystal silicon carbide (0001) plane obtained before ion bombardment or preheating is presented in figure 3. The crystal was in the as-received state after it had been baked out in the vacuum system at 10⁻⁸ pascal. A carbon contamination peak was evident as well as an oxygen peak. The carbon peak in the spectrum was similar to that obtained for amorphous carbon (refs. 5 and 6).

The Auger spectrum taken after the silicon carbide surface had been sputter cleaned clearly revealed the main silicon peak at 91 eV and a carbon peak at 272 eV as well as a small argon peak, as shown in figure 4. The oxygen peak is negligible. Moreover the carbon peak is of the carbide type. The three carbide-type Auger peaks are labeled A₀ to A₂ in figure 4, where A is used to denote an Auger peak.

Surface preheated at 800° C.—The spectra for the silicon carbide surface heated to 250° C with a heating time of 3 hours revealed that carbon Auger

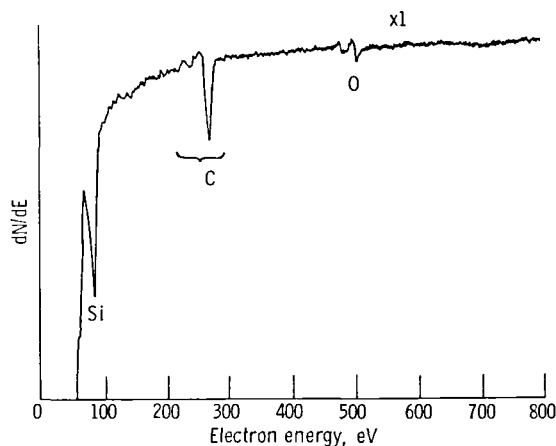


Figure 3. — Auger spectrum of silicon carbide (0001) surface before surface treatment (after bakeout).

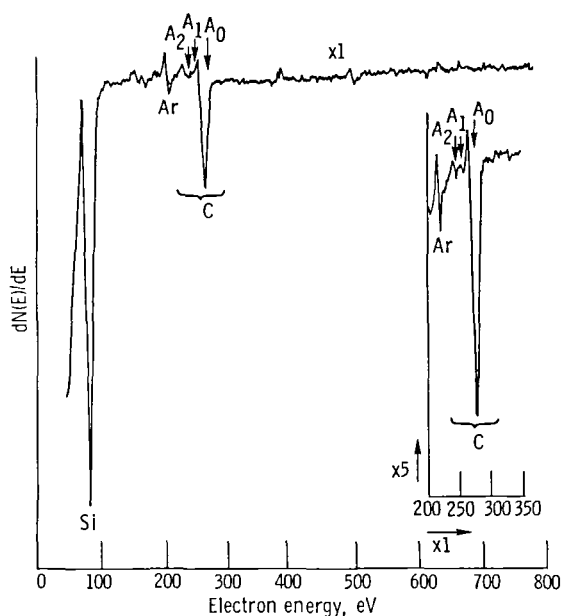


Figure 4. - Auger spectrum of silicon carbide (0001) surface after argon sputter cleaning.

peaks were of the carbide type. Successive heating times of 1 or 3 hours did not make any difference in Auger spectra.

Spectra of the surfaces at room temperature were also taken after heating them to 400°, 550°, 700°, and 800° C with successive heating times of 1 and 3 hours at each temperature. The spectrum presented in figure 5 was taken from the surface after heating it for 3 hours at 800° C in a vacuum of 10^{-8} pascal. Again, the heating time did not make any difference in the Auger spectrum. The spectrum of this surface reveals that the main silicon peak of silicon carbide appears at 88 eV and that the carbon peak, of the carbide type, is at 271 eV (fig. 5). For the various surfaces heated to 250°, 400°, 550°, 700°, and 800° C, the ratio of the carbide type of carbon peak height to the silicon peak height was in the following order: 250° C > 400° C > 550° C > 700° C > 800° C.

The spectra of the silicon carbide surfaces preheated to 250°, 400°, and 550° C revealed that the silicon carbide surfaces contained impurities such as sulfur, oxygen, and nitrogen. On the other hand, the spectrum of the surface preheated to 800° C was the same as that obtained for an argon-sputter-cleaned surface, as indicated by figure 4. After being heated to 800° C and cooled to room temperature the silicon carbide specimen was next reheated to 800° C. Silicon carbide surfaces after reheating at any temperature revealed the same Auger spectrum as a surface after preheating to 800° C, as shown in figure 5.

As is generally known, the Auger spectrum of an element strongly depends on the chemical binding state. Therefore the surfaces preheated to 800° C have nearly the same surface condition as those of specimens reheated to 800° C. Friction experiments conducted on the reheated surfaces of silicon carbide are described in the next section.

Surface preheated at 1500° C.—A typical spectrum of the silicon carbide surface after preheating to 1500° C is shown in figure 6. The silicon peak is negligibly small and was difficult to detect by Auger electron spectroscopy. The carbon peak, of the graphite type, is at 271 eV. The graphite type is characterized by a step. The position of the step in the carbon peak is labeled A.

The carbide type of Auger peak changed to the graphite type at about 900° to 1000° C. The silicon peak decreased in size with an increase in temperature. The graphite type of Auger peak, however, increased.

After being heated to 1500° C the silicon carbide specimen was cooled to room temperature and then reheated to 1200° C. The surface of the silicon carbide after reheating at any temperature revealed the same AES spectrum as that shown in figure 6. Friction experiments were conducted on the reheated surface of silicon carbide.

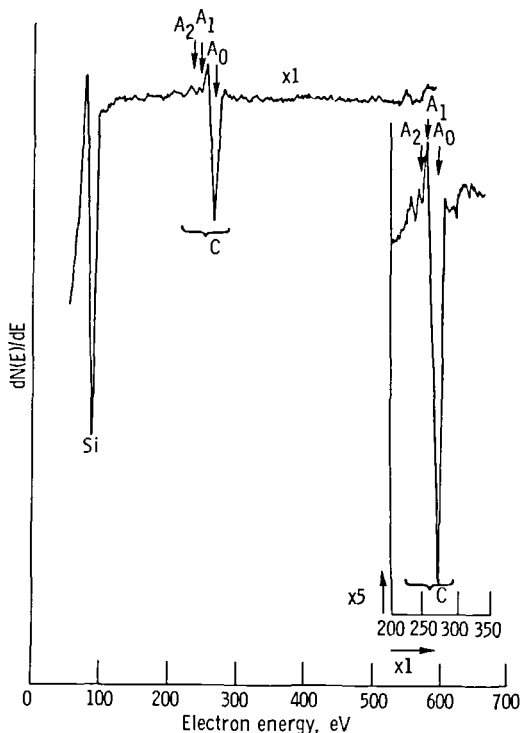


Figure 5. - Auger spectrum of silicon carbide surface after preheating at 800° C for 3 hr in a vacuum of 10^{-8} Pa.

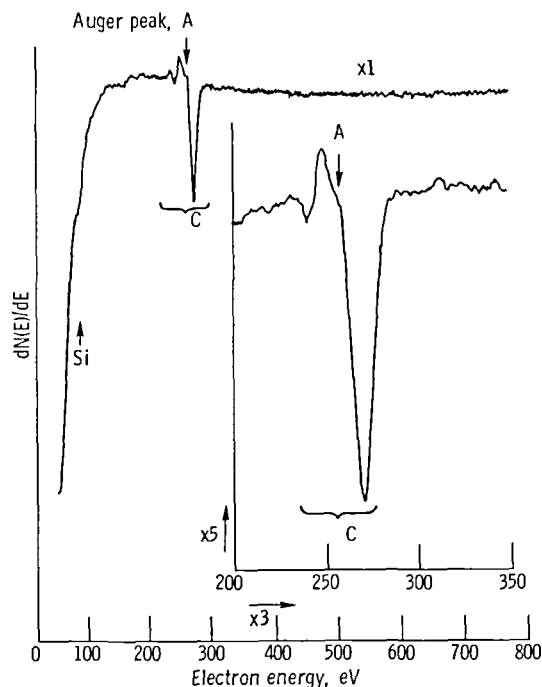


Figure 6. - Auger spectrum of silicon carbide surface after preheating at 1500° C for 3 hr in a vacuum of 10^{-8} Pa.

The silicon carbide surface dissociated at 1500° C, with preferential evaporation of silicon from the silicon carbide near 1500° C. The mechanism for graphite formation according to Badami is that two successive carbon layers form on the surface of silicon carbide after evaporation of the silicon and then collapse into one layer of carbon hexagons with the unit mesh parallel to that of silicon carbide (ref. 6). This layer of hexagons with a *c-c* distance of 0.185 nanometer shrinks to a graphite layer with a *c-c* distance of 0.142 nanometer (ref. 6). Van Bommel, *et al.*, concluded that there is a different graphitization behavior for each of the two polar faces (ref. 7). In the initial state the silicon face (already mentioned in the section Materials) yields a monocrystalline graphite layer (ref. 7), and the carbon face yields a polycrystalline graphite layer. The collapse of the carbon of three successive silicon carbide layers is the most probable mechanism for the initial stages of the graphitization of silicon carbide basal surfaces.

The Auger peaks of carbon provide valuable information about the form in which the carbon is present on the surface of the silicon carbide and serve as a fingerprint for identifying carbon compounds. The Auger results correlated with the results of Van Bommel, *et al.* (ref. 7).

Friction Behavior and Its Crystallographic Anisotropy

Sliding friction experiments were conducted with single-crystal silicon carbide in contact with iron in vacuum. Figure 7 shows a typical friction-force trace resulting from such sliding. The line *a-b* in figure 7 represents the region where both loading and tangential forces were being applied but where no gross sliding occurred. At point *b* the onset of slip occurred. After reaching point *b* the surfaces of iron and silicon carbide were in slip. All coefficients of friction reported are static measurements. (The coefficient of static friction μ is defined as $\mu = \bar{F}/W$, where \bar{F} is the friction force at which the first break is observed in the friction force trace and W is the normal load.)

Friction experiments were conducted with the iron rider sliding on the silicon carbide after various times in static contact, before sliding, at a temperature of 800° C. The coefficient of friction depended considerably on the time in contact. The coefficient of friction increased with time and reached some equilibrium value after approximately 20 seconds in contact. After about 3 minutes in contact, however, the coefficient of friction started again to increase and then again reached some equilibrium value at approximately 5 minutes, after which very little change in the coefficient of friction was observed. The characteristics shown in figure 8 may be due to creep, as a result of which the area of contact increased with loading time. There may also be a time factor involved in diffusion across the interface,

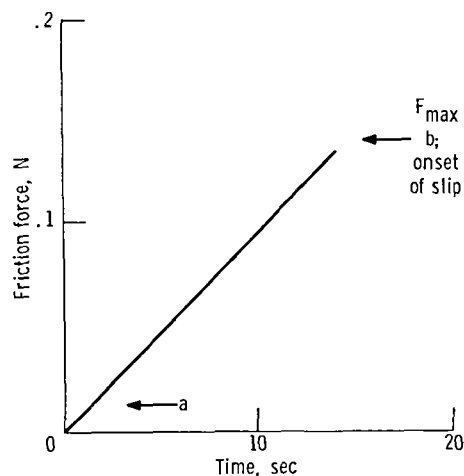


Figure 7. - Friction force curve for iron rider sliding on single-crystal silicon carbide {0001} surface preheated to 800° C. Normal load, 0.2 N; sliding temperature, 800° C; vacuum, 10^{-8} Pa.

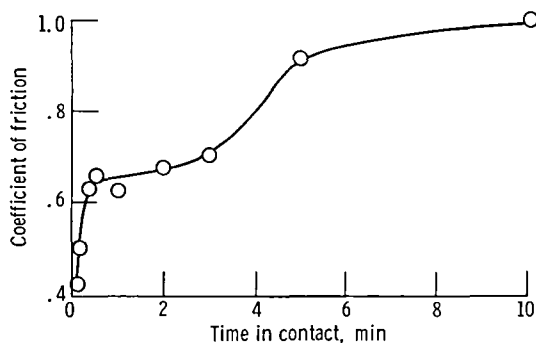


Figure 8. - Effect of time in contact on coefficient of friction for iron rider sliding in $\langle 11\bar{2}0 \rangle$ direction on single-crystal silicon carbide surface preheated to 800°C . Normal load, 0.2 N; sliding temperature, 800°C ; vacuum, 10^{-8} Pa.

which leads to a strengthening of the junctions with contact time (ref. 11). It is very possible that recrystallization at high temperature introduces grain boundaries in the iron surfaces. This may account for the observed increase in the coefficient of friction and the junction strengthening with time in contact (ref. 12). Hence, for the adhesion and friction experiments in succeeding sections, the time in contact before sliding was held at 30 seconds in order to maintain the same experimental conditions.

Silicon carbide (0001) surfaces that were argon sputter cleaned and brought into contact with iron revealed no significant change in the coefficient of friction with loads of 0.05 to 0.3 newton (ref. 4). The average coefficients of friction were 0.48 in the $\langle 10\bar{1}0 \rangle$ direction and 0.44 in the $\langle 11\bar{2}0 \rangle$ direction. The friction traces were primarily characterized by marked stick-slip behavior (refs. 4 and 5). This type of friction is expected where strong adhesion occurs at the interface. Data were obtained for iron sliding on a single-crystal silicon carbide (0001) surface.

The frictional properties of the silicon carbide (0001) surface in contact with iron at temperatures to 800°C after preheating to 800°C are indicated by the data in figure 9. The silicon carbide (0001) surface was heated to 800°C before the friction experiments were begun. The coefficient of friction generally increased with increasing temperature from about 0.5 in the $\langle 10\bar{1}0 \rangle$ sliding direction and 0.4 in the $\langle 11\bar{2}0 \rangle$ sliding direction at room temperature to 0.75 and 0.63, respectively, at 800°C . Although the coefficient of friction remained low below 300°C , it increased rapidly with increasing temperature in the range 300° to 600°C , but there was little further increase in friction above 600°C .

The rapid increase in friction at temperatures from 300° to 600°C may be due to an increase in the adhesion resulting from recrystallization of iron.

Iron normally recrystallizes at 200° to 300°C . The experiments herein were started with textured surfaces of polycrystalline iron (ref. 12). Iron in contact with silicon carbide will recrystallize at 300° to 600°C . Recrystallized-annealed iron surfaces are less resistant to deformation and adhesion than are textured surfaces (ref. 12). The general increases in friction at high temperatures are due to increased adhesion and increased plastic flow at the area of contact.

The data in figure 9 indicate that the friction behavior of silicon carbide in contact with iron is highly anisotropic over the entire temperature range from room temperature to 800°C . Several slip systems have been observed in α -silicon carbide, including the (0001) $\langle 11\bar{2}0 \rangle$, (3301) $\langle 11\bar{2}0 \rangle$, and (1010) $\langle 11\bar{2}0 \rangle$ (refs. 13 and 14). The preferred crystallographic slip direction, or the direction of shear for the basal (0001) plane, is the $\langle 11\bar{2}0 \rangle$ direction. Examination of the coefficient of friction on the basal plane indicates that it was lower in the $\langle 11\bar{2}0 \rangle$ direction than in the $\langle 10\bar{1}0 \rangle$ direction. That is, it was lower in the preferred crystallographic direction than in the $\langle 10\bar{1}0 \rangle$ direction.

The friction properties of silicon carbide (0001) surfaces in contact with iron at temperatures to 1200°C are shown in figure 10. The silicon carbide

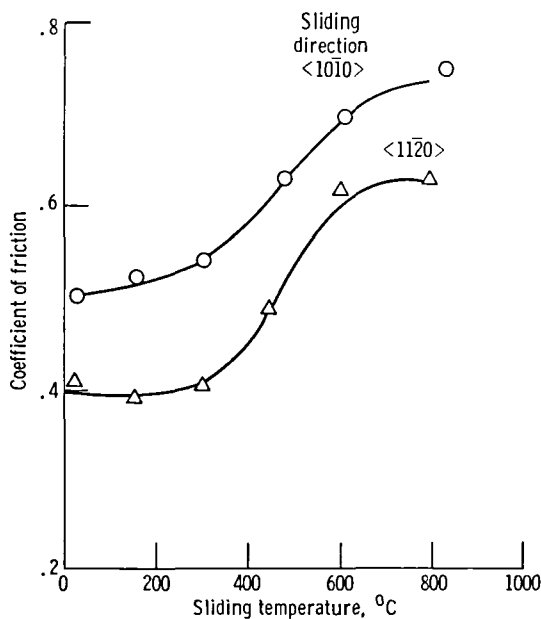


Figure 9. - Effect of sliding temperature and crystallographic orientation on coefficient of friction for single-crystal silicon carbide surface preheated to 800°C sliding against iron rider. Normal load, 0.2 N; vacuum, 10^{-8} Pa.

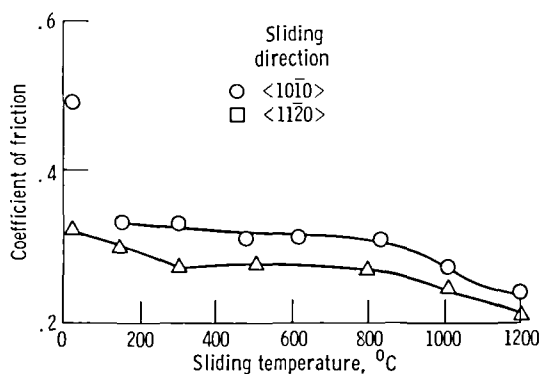


Figure 10. - Effect of sliding temperature and crystallographic orientation on coefficient of friction for single-crystal silicon carbide (0001) surface preheated to 1500° C sliding against iron rider. Normal load, 0.2 N; vacuum, 10^{-8} Pa.

(0001) surfaces were first heated to 1500° C for 2 hours in a vacuum of 10^{-8} pascal before the friction experiments were begun. The silicon peak was nearly gone from the spectrum. When compared with the results of figure 9, the coefficients of friction are generally lower in figure 10, with the exception of the coefficient of friction at room temperature in the $\langle 1010 \rangle$ sliding direction on the (0001) surface.

The low friction at the high temperatures appears to correlate with the graphitization of the silicon carbide surface. The coefficients of friction on this surface at the high temperatures are nearly consistent with those on pyrolytic graphite in sliding contact with single-crystal iron in a vacuum of 10^{-8} pascal (ref. 15).

The formation of graphite hexagons is possible, and an orientation of the formed graphite layer on the silicon carbide surface that was preheated to 1500° C seems probable (fig. 10). This orientation effect influences the friction behavior of the basal plane of silicon carbide.

In the results presented in figure 10, the coefficient of friction in the $\langle 1010 \rangle$ direction at room temperature is an exception. The coefficient of friction at these experimental conditions is almost the same as that on the surface, with silicon and carbon of the carbide type present at room temperature (fig. 9). Seven measurements were conducted with the same experimental conditions to examine the coefficients of friction at a load of 0.2 newton. The coefficients of friction were divided into two groups: One, which was obtained by averaging five data points, is 0.48, and the other, obtained by averaging two data points, is 0.39. The deviation in each group was 3 to 4 percent of each average value. In figure 10 only the coefficient of friction 0.48 is plotted. This

point may have resulted from a breaking up of the graphite layer during static contact and the sliding of the iron rider against the silicon carbide. It is possible that the iron breaks through the graphite layer and comes directly in contact with the silicon carbide. When this occurs there can be very strong adhesive bonding at the sliding interface. The low coefficient of friction, 0.39, appears to correlate with the graphitization of the silicon carbide surface.

The friction behavior of a silicon carbide surface in contact with iron as a function of normal load at room temperature is shown in figure 11. The silicon carbide surface was preheated to 1500° C. The coefficient of friction did not depend on the normal load in the load range 0.1 to 0.25 newton. The coefficient of friction, however, remained low at a load of about 0.05 newton. The low coefficient of friction at 0.05 newton is the same value as that shown at high temperatures in figure 10. At the higher loads of 0.1 to 0.25 newton the graphite layer covering the silicon carbide surface was easily broken by the sliding action, and the friction was higher. At the lower load of 0.05 newton, however, the graphite layer was not broken by the sliding action of iron, and the friction was accordingly lower.

Lastly, the friction traces with the silicon carbide-iron couples, shown in figures 9 and 10, reveal a clear difference in behavior. The friction traces with silicon carbide that was preheated to 800° C are characterized by stick-slip behavior (fig. 12(a)). On the other hand the friction traces with silicon carbide that was preheated to 1500° C are characterized by smooth fluctuating behavior with no evidence of

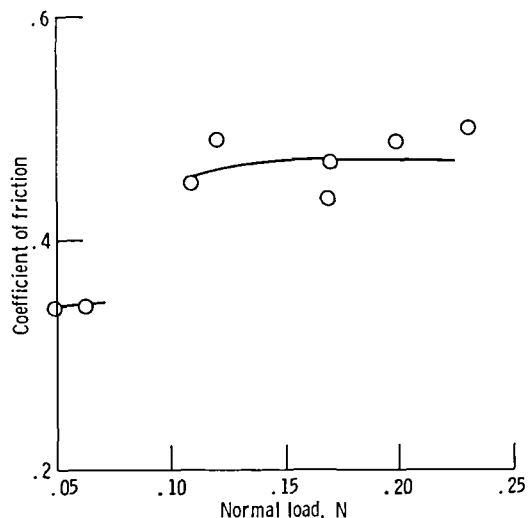


Figure 11. - Effect of load on coefficient of friction for single-crystal silicon carbide surface preheated to 1500° C sliding against iron rider. Sliding direction, $\langle 1010 \rangle$; sliding temperature, room; vacuum, 10^{-8} Pa.

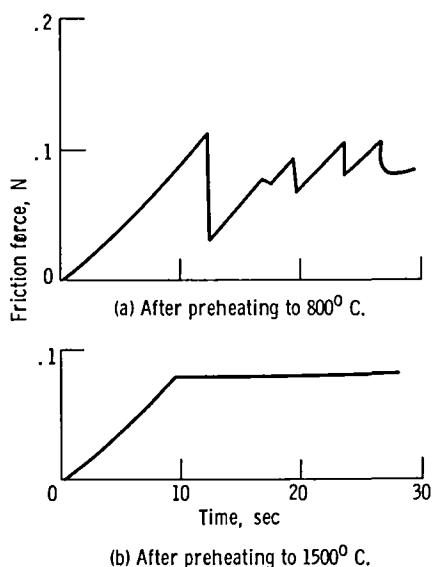


Figure 12. - Typical friction force traces for iron rider sliding in $\langle 10\bar{1}0 \rangle$ direction on single-crystal silicon carbide (0001) surface. Normal load, 0.2 N; sliding temperature, room; vacuum, 10^{-8} Pa.

stick-slip (fig. 12(b)). This difference is due to the effect of the graphite layer. The kinetic friction properties of silicon carbide in sliding contact with iron were almost the same as those shown in figures 8 to 11.

Summary of Results

As a result of sliding friction experiments conducted in this investigation with single-crystal silicon carbide preheated to 800° and 1500° C, the following conclusions were drawn:

1. Surfaces of silicon carbide that were preheated to 800° C or were bombarded with argon ions revealed the main silicon peak and a carbide type of carbon peak in the Auger spectra.

Surfaces of silicon carbide that were preheated to 1500° C revealed only a graphite type of carbon peak in the Auger spectra. The silicon peak diminished to the point where it was barely detectable.

2. The surfaces of silicon carbide that were preheated to 800° C gave 1.5 times to 3 times higher coefficients of friction than did silicon carbide surfaces preheated to 1500° C.

3. The coefficient of friction was markedly dependent on crystallographic orientation: It was lower in the $\langle 11\bar{2}0 \rangle$ direction than in the $\langle 10\bar{1}0 \rangle$ direction. That is, the coefficient of friction was lower in the preferred crystallographic direction of slip.

4. Preheating the surface to 800° C produced stick-slip behavior. Preheating the surface to 1500° C produced smooth friction traces without any stick-slip behavior.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 20, 1980,
506-53.

References

1. O'Connor, J. R.; and Smiltens, J., eds.: *Silicon Carbide, a High Temperature Semiconductor*. Pergamon Press, 1960.
2. Marshall, R. C.; Faust, J. W., Jr.; and Ryan, C. E.: *Silicon Carbide—1973, Proceedings of the Third International Conference*. University of South Carolina Press, 1974.
3. Komanduri, R.; and Shaw, M. C.: *Attritious Wear of Silicon Carbide*. *J. Eng. Ind.*, vol. 98, no. 4, Nov. 1976, pp. 1125-1134.
4. Miyoshi, K.; and Buckley, D. H.: *Friction and Wear Behavior of Single-Crystal Silicon Carbide in Sliding Contact with Various Metals*. *ASLE Trans.*, vol. 22, no. 3, 1979, pp. 245-256.
5. Miyoshi, K.; and Buckley, D. H.: *Effect of Oxygen and Nitrogen Interactions on Friction of Single-Crystal Silicon Carbide*. NASA TP-1265, 1978.
6. Badami, D. V.: *X-Ray Studies of Graphite Formed by Decomposing Silicon Carbide*. *Carbon*, vol. 3, no. 1, 1965, pp. 53-57.
7. Van Bommel, A. J.; Crombeen, J. E.; and Van Tooren, A.: *LEED and Auger Electron Observations of the SiC (0001) Surface*. *Surf. Sci.*, vol. 48, no. 2, 1975, pp. 463-472.
8. Taylor, A.; and Laidler, D. S.: *Formation and Crystal Structure of Silicon Carbide*. *Brit. J. Appl. Phys.*, vol. 1, no. 7, July 1950, pp. 174-181.
9. Hull, A. W.: *The Crystal Structure of Carborundum*. *Phys. Rev.*, vol. 13, Apr. 1919, pp. 292-295.
10. Ott, H.: *Structure of Carborundum (SiC)*. *Z. Krist.*, vol. 61, 1925, pp. 515-531; *The Lattice of Carborundum Type I*. *Z. Krist.*, vol. 62, 1926, pp. 201-217.
11. Bowden, F. P.; and Tabor, D.: *The Friction and Lubrication of Solids*. Part II. Clarendon Press (London), 1964.
12. Buckley, D. H.: *Friction, Wear, and Lubrication in Vacuum*. NASA SP-277, 1971.
13. Amelinckx, S.; Strumane, G.; and Webb, W. W.: *Dislocations in Silicon Carbide*. *J. Appl. Phys.*, vol. 31, no. 8, Aug. 1960, pp. 1359-1370.
14. Brookes, C. A.; O'Neill, J. B.; and Redfern, B. A. W.: *Anisotropy in the Hardness of Single Crystals*. *Proc. R. Soc. (London)*, Ser. A, vol. 322, no. 1548, Mar. 23, 1971, pp. 73-88.
15. Buckley, Donald H.; and Brainard, William A.: *Friction and Wear of Metals in Contact with Pyrolytic Graphite*. *Carbon*, vol. 13, 1975, pp. 501-508. (Also NASA TM-78322, 1975.)

1. Report No. NASA TP-1756	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle CHANGES IN SURFACE CHEMISTRY OF SILICON CARBIDE (0001) SURFACE WITH TEMPERATURE AND THEIR EFFECT ON FRICTION		5. Report Date November 1980	
		6. Performing Organization Code	
7. Author(s) Kazuhisa Miyoshi and Donald H. Buckley		8. Performing Organization Report No. E-475	
		10. Work Unit No. 506-53	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135		11. Contract or Grant No.	
		13. Type of Report and Period Covered Technical Paper	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		14. Sponsoring Agency Code	
15. Supplementary Notes			
16. Abstract Friction studies were conducted with a silicon carbide (0001) surface contacting polycrystalline iron. The surface of silicon carbide was pretreated (1) by bombarding it with argon ions for 30 minutes at a pressure of 1.3 pascals, (2) by heating it at 800° C for 3 hours in vacuum at a pressure of 10 ⁻⁸ pascal, or (3) by heating it at 1500° C for 3 hours in a vacuum of 10 ⁻⁸ pascal. Auger emission spectroscopy was used to determine the presence of silicon and carbon and the form of the carbon. The surfaces of silicon carbide bombarded with argon ions or preheated to 800° C revealed the main Si peak and a carbide type of C peak in the Auger spectra. However, the surfaces preheated to 1500° C revealed only a graphite type of C peak in the Auger spectra, and the Si peak had diminished to a barely perceptible amount. The surfaces of silicon carbide preheated to 800° C gave a 1.5 to 3 times higher coefficient of friction than did the surfaces of silicon carbide preheated to 1500° C. The coefficient of friction was lower in the <1120> direction than in the <1010> direction; that is, it was lower in the preferred crystallographic slip direction.			
17. Key Words (Suggested by Author(s)) Silicon carbide Auger High temperature Friction		18. Distribution Statement Unclassified - unlimited STAR Category 27	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 11	22. Price* A02

* For sale by the National Technical Information Service, Springfield, Virginia 22161

NASA-Langley, 1980

~ ~

~ ~

National Aeronautics and
Space Administration

Washington, D.C.
20546

Official Business

Penalty for Private Use, \$300

SPECIAL ~~FOURTH~~ CLASS MAIL
BOOK

Postage
Nation
Space
NASA



U.S. MAIL



POSTMASTER: If Undeliverable (Section 158
Postal Manual) Do Not Return
